# Estimating Organic Carbon from Loss-On-Ignition in Northern Arizona Forest Soils

### Scott R. Abella\*

Public Lands Inst. and School of Life Sciences Univ. of Nevada-Las Vegas 4505 S. Maryland Pkwy. Las Vegas, NV 89154-2040

### Brian W. Zimmer

Dep. of Geology Northern Arizona Univ. Flagstaff, AZ 86011-4099

Many studies in ecology, soil science, and global climate change require accurate estimates of soil organic C (SOC). When calibrated with direct SOC determinations, loss-on-ignition (LOI) has been proposed as a rapid, inexpensive, and accurate method for estimating SOC. We collected 0- to 15- and 15- to 50-cm mineral soil samples from 102 plots within a 110000-ha ponderosa pine (Pinus ponderosa P. & C. Lawson) landscape to develop regression equations between LOI and SOC measured with an elemental C analyzer. We tested nine LOI temperature-duration combinations ranging from 300 to 600°C and 2 to 6 h to discern optimal combinations for estimating SOC, used the optimal combination to develop regressions for 100 samples each of 0- to 15- and 15- to 50-cm depths, and assessed whether stratifying samples into ecosystem types improved LOI–SOC equations. Pearson  $r^2$  values between LOI and SOC did not exceed 0.74 for any LOI temperature-duration combination. These values showed no consistent trend to change with increasing duration, but tended to be slightly higher at the lowest temperature (300°C). Multiple regressions, including LOI and clay concentration, explained only 78 (0-15 cm) and 64% (15-50 cm) of the variation in SOC. Relationships between LOI and SOC found in this study are among the weakest reported in the soil literature, and it remains unclear precisely why observed relationships were weak. Our results suggest that LOI may be useful for roughly estimating SOC in this region, but other methods or modifications to LOI are needed when more accurate SOC measurements are required.

Abbreviations: LOI, loss-on-ignition; SOC, soil organic carbon; SOM, soil organic matter.

Estimates of SOC often are needed for applications in ecology, soil science, and global climate change. Many methods are available for measuring SOC, each with advantages and disadvantages in terms of accuracy, expense, and convenience (Nelson and Sommers, 1996). For example, elemental C analyzers are accurate, but they may be expensive to purchase, operate, and maintain. In comparison, the Walkley–Black procedure is cheap and fast to perform, but exhibits variable SOC recovery and generates hazardous byproducts such as Cr.

Loss-on-ignition, which involves combusting samples at high temperatures and measuring weight loss, has been proposed as an inexpensive, convenient, and accurate method for estimating SOC (Ball, 1964; Lowther et al., 1990; Konen et al., 2002). In this method, LOI is used as an independent variable in regression equations to estimate SOC, which has been measured on test samples using direct methods such as elemental C analyzers (David, 1988; Cambardella et al., 2001; Konen et al., 2002). Strong linear relationships between LOI and SOC have been reported, with  $r^2$  values often exceeding 0.90 (Christensen and Malmros, 1982; Soon and Abboud

doi:10.2136/sssaj2006.0136

\*Corresponding author (scott.abella@unlv.edu).

1991; Cambardella et al., 2001). For example, Lowther et al. (1990) reported an  $r^2$  of 0.99 for an LOI–SOC regression in Australian radiata pine (*Pinus radiata* D. Don) forest soils. More recently, Konen et al. (2002) reported  $r^2$  values of 0.94 to 0.98 in north-central USA soils.

While these studies suggest that LOI has great potential for easily and accurately estimating SOC, several important considerations accompany LOI. Loss-on-ignition does not necessarily represent soil organic matter (SOM), because LOI can decompose inorganic constituents (e.g., minerals and clay structural water) while not all SOM ignites (Alexander and Byers, 1932; Ball, 1964; Schulte and Hopkins, 1996). Optimal heating temperatures and durations to maximize SOM combustion, while minimizing inorganic combustion, are difficult to determine. However, these temperatures and durations can substantially affect LOI and SOC/LOI ratios (Keeling, 1962; Ben-Dor and Banin, 1989; Schulte et al., 1991). As with SOM, the ratio of SOC to LOI also varies among soil types and with depth within a soil (Broadbent, 1953; Howard, 1965; Christensen and Malmros, 1982). As a result, numerous researchers have suggested that LOI-SOC equations be developed separately for different soil depths and types (Spain et al., 1982; David, 1988; Konen et al., 2002). Additionally, both SOM and SOC have been estimated using LOI, making comparisons troublesome in the literature (Konen et al., 2002). Several researchers have suggested that because of the difficulty of accurately measuring SOM, and more recently with increased interest in C cycling, LOI generally should be used to estimate SOC rather than SOM (e.g., Broadbent, 1953; Nelson and Sommers, 1996; Schulte and Hopkins, 1996). If desired, SOC can be converted to SOM using SOC/SOM ratios determined on test

Soil Sci. Soc. Am. J. 71:545-550

Received 29 Mar. 2006.

<sup>©</sup> Soil Science Society of America

<sup>677</sup> S. Segoe Rd. Madison WI 53711 USA

All rights reserved. No part of this periodical may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopying, recording, or any information storage and retrieval system, without permission in writing from the publisher. Permission for printing and for reprinting the material contained herein has been obtained by the publisher.

samples or obtained from the literature (Schulte and Hopkins, 1996; Cambardella et al., 2001).

Most LOI research in North America has been performed on eastern, central, or northwestern temperate prairie or forest soils (e.g., Ranney, 1969; Schulte et al., 1991; Konen et al., 2002). Our objective was to develop regression equations that estimate SOC (measured directly with an elemental C analyzer) from LOI for forest soils on a semiarid northern Arizona ponderosa pine landscape. Current research in these forests focuses on developing and evaluating ecological restoration and management prescriptions to reduce risks of wildfire and improve ecosystem health (Covington et al., 1997; Allen et al., 2002). If SOC can be accurately estimated from relatively inexpensive and convenient LOI, this could be useful for ecological studies and for monitoring management effects on SOC (Gundale et al., 2005). Our specific objectives were to: (i) test effects of heating temperature and duration on LOI and LOI-SOC relationships to determine optimal heating procedures for estimating SOC from LOI; (ii) develop regional regression equations to estimate SOC from LOI using these optimal heating temperatures and durations; and (iii) examine the variability of LOI-SOC relationships within and among ecosystem types of similar vegetation and soils previously classified in an ecosystem classification.

## MATERIALS AND METHODS Study Area

We collected soil samples at elevations between 1920 and 2660 m within a 110 000-ha landscape in the northern half of the Coconino National Forest and in the Northern Arizona University Centennial Forest. This landscape surrounds the City of Flagstaff in northern Arizona (study area SW corner 35°04' N, 111°53' W; NW corner 35°29' N, 111°51' W; NE corner 35°23' N, 111°31' W; SE corner 35°01' N, 111°23' W). Based on three weather stations, mean total precipitation across the study area ranges from 42 to 56 cm yr<sup>-1</sup>, snowfall from 152 to 233 cm yr<sup>-1</sup>, and mean maximum daily temperatures from 15.7 to 17.5°C (Western Regional Climate Center, Reno,

NV). Prevalent soil parent materials include volcanic cinders, basalt, benmoreite, and limestone. Major soil subgroups are Typic and Udic Argiborolls, Typic and Mollic Eutroboralfs, Typic Ustorthents, and Vitrandic Ustochrepts (Miller et al., 1995). Forests are primarily pure ponderosa pine, but trembling aspen (*Populus tremuloides* Michx.) or Gambel oak (*Quercus gambelii* Nutt.) occasionally occur with ponderosa pine. Understories are grass dominated, with major species including squirreltail [*Elymus elymoides* (Raf.) Swezey], mountain muhly [*Muhlenbergia montana* (Nutt.) A.S. Hitchc.], Arizona fescue (*Festuca arizonica* Vasey), and blue grama [*Bouteloua gracilis* (Willd. ex Kunth) Lag. ex Griffiths].

### Sample Collection

On 102, 0.05-ha (20- by 25-m) plots distributed across the landscape, we collected soil samples of 0- to 15- and 15- to 50-cm depths from two pits per plot. We composited samples for each depth on a plot basis ( $\sim$ 2 kg of field-moist soil from each depth of each plot). Plots were located within 10 ecosystem types previously classified in a landscape ecosystem classification for the study area (Abella and Covington, 2006). Ecosystem classification groups sites into ecosystem types, each internally similar in topographic, soil, and vegetative characteristics (Barnes et al., 1982). We stratified our sampling by ecosystem type in the present study because sharp differences in several soil properties occurred among ecosystem types (Table 1). This stratification is similar to a recent LOI study by Konen et al. (2002), who found that LOI–SOC regression equations differed among land resource areas in the north-central USA.

### Laboratory Analysis

Samples were air dried and sieved through a 2-mm sieve. We analyzed samples for texture (hydrometer method) and pH (1:2 soil/  $0.01 \ M \ CaCl_2$ ) following Sparks (1996) and Dane and Topp (2002). We also estimated  $CaCO_3$  equivalent using an approximate gravimetric method (Goh et al., 1993). After grinding samples to pass a 53-µm sieve and pretreating samples with 10% HCl to remove potential carbonates, we analyzed 30-mg samples for organic C using an elemental C analyzer (FlashEA 1112, Thermo Electron Corp., Waltham, MA).

Table 1. Summary of soil properties of ponderosa pine ecosystems classified by Abella and Covington (2006) within a 110 000-ha northern Arizona landscape.

<b>D</b> (	Depth	Ecosystem type (no. of plots)†									
Property		1(6)	2(6)	3(6)	4(6)	5(18)	6(13)	7(23)	8(12)	9(6)	10(6)
	cm										
Texture‡	0-15	S	sl	cl	sl	sl	I	Ι	sil	Ι	cl
	15-50	S	sl	С	scl	cl	cl	cl	I	I	cl
рН	0-15	$6.5\pm0.2\$$	$6.6 \pm 0.1$	$6.6\pm0.3$	$6.9\pm0.2$	$6.1 \pm 0.3$	$6.3 \pm 0.3$	$6.0 \pm 0.1$	$6.1 \pm 0.2$	$6.4 \pm 0.2$	$5.9 \pm 0.2$
	15-50	$6.7 \pm 0.1$	$6.8 \pm 0.1$	$6.8\pm0.3$	$7.0 \pm 0.1$	$6.4\pm0.5$	$6.3 \pm 0.3$	$6.0\pm0.2$	$6.2 \pm 0.2$	$6.2 \pm 0.2$	$6.1\pm0.0$
CaCO <sub>3</sub> , %¶	0-15	<1	<1	<1	$3 \pm 6$	<1	<1	<1	<1	<1	<1
	15-50	<1	<1	<1	$28 \pm 55$	2 ± 2	<1	<1	<1	$2 \pm 4$	<1
Organic C, %	0-15	$1.5 \pm 0.9$	$2.1\pm0.5$	$1.3 \pm 0.2$	$1.1 \pm 0.3$	$1.3 \pm 0.5$	$1.3 \pm 0.3$	$1.9\pm0.5$	$2.2\pm0.7$	$3.1 \pm 0.9$	$1.6 \pm 0.3$
	15-50	$0.3 \pm 0.1$	$0.9 \pm 0.3$	$1.0 \pm 0.4$	$0.9 \pm 0.3$	$0.8 \pm 0.2$	$1.0 \pm 0.2$	$1.1 \pm 0.4$	$1.2 \pm 0.5$	$1.7 \pm 0.4$	$1.0 \pm 0.2$
LOI, %#	0-15	$0.9 \pm 0.1$	$3.2 \pm 0.8$	$3.0 \pm 0.2$	$2.5 \pm 0.5$	$2.8 \pm 0.7$	$2.3 \pm 0.3$	$4.2 \pm 0.9$	$4.2 \pm 0.9$	$6.5 \pm 1.7$	$3.8 \pm 0.5$
	15-50	$0.7 \pm 0.6$	$2.2 \pm 0.4$	$2.9\pm0.4$	$2.7\pm0.7$	$2.6\pm0.7$	$2.6\pm0.6$	$3.4 \pm 0.5$	$2.8\pm0.9$	$3.8 \pm 0.7$	$3.1 \pm 0.3$

+ 1 = black cinders/Phacelia, 2 = red cinders/Bahia, 3 = clay basalt/Gutierrezia, 4 = xeric limestone/Bouteloua, 5 = mesic limestone/mixed flora, 6 = xeric basalt/Muhlenbergia, 7 = rocky basalt/Sporobolus, 8 = mesic basalt/Festuca, 9 = aspen/Lathyrus, and 10 = park/Symphyotrichum.

 $\pm$  c = clay, cl = clay loam, l = loam, s = sand, scl = sandy clay loam, sl = sandy loam, sil = silt loam.

§ Mean  $\pm$  standard deviation.

¶ CaCO<sub>3</sub> equivalent estimated using an approximate gravimetric method (Goh et al., 1993).

# Loss-on-ignition (300°C, 2 h).

Based on analysis of duplicate samples every 10 samples, measurement error averaged  $\leq$ 5% for all analyses. Analysis of laboratory reference standards for the C analyzer also differed on average by <0.1% C from true values, and C was not recorded in blanks.

Separately for 0- to 15- and 15- to 50-cm depths, we randomly selected three plots in each of the 10 ecosystem types from which to extract samples to test temperature and duration effects on LOI and correlations with SOC (n = 30 for each depth). We heated sieved (<2 mm), 5-g samples (air-dry weight) from each plot at 105°C for 24 h in an electric oven and then weighed the samples to measure moisture loss before LOI. Loss-on-ignition treatments were applied factorially, with three levels each of temperature (300, 450, and 600°C) and duration (2, 4, and 6 h). These temperature and duration combinations span a range commonly used in LOI studies (Schulte and Hopkins, 1996). We placed samples in completely random order in individual porcelain crucibles and performed LOI treatments in a Thermolyne model F-A1730 muffle furnace (Sybron Corp., Dubuque, IA). The internal chamber dimensions of the furnace were 25 (width), 23 (height), and 35 (depth) cm. Based on analysis of three randomly selected duplicate samples for each temperature-duration combination, LOI repeated measurement errors averaged 3.5% for 0- to 15-cm and 3.9% for 15- to 50-cm samples. We also examined the effects on LOI of shorter time durations (15, 30, 60, 90, and 120 min) at 300°C using 0- to 15-cm samples from three randomly selected plots in each of 10 ecosystem types.

Based on LOI–SOC correlations found in the factorial experiment of LOI temperature–duration combinations, we selected 300°C and 2 h as the optimal combination for estimating SOC from LOI. Correlations on average were highest at 300°C, and since they changed little with increasing duration, we selected 2 h as more convenient than longer durations. We used this combination to develop regression equations for estimating SOC from LOI using samples from 100 plots for each of the 0- to 15- and 15- to 50-cm depths. A maximum of 50 samples could be placed in our muffle furnace at one time, so we randomly selected 100 of our 102 plots for inclusion in the analysis and heated samples in sets of 50 for each depth.

### **Statistical Analysis**

We analyzed the factorial temperatureduration experiment with LOI weight as the response variable using a two-factor analysis of variance with plots serving as blocks. Plots were designated as blocks because samples for treatment were extracted from composite plot samples. Tukey's test was used for mean separation. Raw data met equal variance (Levene test) and normality (Shapiro-Wilk W test) assumptions. Pearson  $r^2$  values between LOI and SOC also were computed for each temperature-duration combination. We developed LOI-SOC regression equations based on 300°C, 2-h LOI using simple linear regression. Including soil texture with LOI has occasionally been reported to enhance estimation of SOC (Spain et al., 1982; Donkin, 1991; Soon and Abboud, 1991). To examine whether texture or other measured variables improved estimation, we used forward, stepwise multiple regression, inputting LOI, sand percentage, clay percentage, pH, and CaCO<sub>3</sub> equivalent. Statistical analyses were performed using SAS JMP (SAS Institute, 2002).

# Table 2. Mean loss-on-ignition (%) and *r*<sup>2</sup> values with organic C for different temperature and duration combinations for northern Arizona ponderosa pine forest soils.

	Temperature						
Duration	300°C	450°C	600°C				
h		%					
		<u>0–15 cm</u>					
2	3.9 ± 1.8a (0.66)†	$5.8 \pm 2.5b \ (0.56)$	$6.4 \pm 2.7 c \ (0.48)$				
4	3.6 ± 1.6a (0.69)	$5.5 \pm 2.4b \ (0.64)$	$6.4 \pm 2.6c \ (0.45)$				
6	3.9 ± 1.8a (0.74)	$5.5 \pm 2.6b \ (0.62)$	6.5 ± 2.7c (0.52)				
		<u>15–50 cm</u>					
2	2.9 ± 1.1a (0.61)	$4.8 \pm 1.9b \ (0.58)$	5.6 ± 2.2c (0.53)				
4	3.1 ± 1.2a (0.61)	$4.5 \pm 1.9b \ (0.62)$	$5.4 \pm 2.0c \ (0.59)$				
6	3.1 ± 1.3a (0.64)	$4.8 \pm 1.9b \ (0.56)$	5.4 ± 2.1c (0.55)				

+ Values are mean loss-on-ignition  $\pm$  standard deviation (Pearson  $r^2$  with organic C). Means within depths (0–15 or 15–50 cm) without shared letters differ at P < 0.05 (Tukey's test).

### RESULTS

In the factorial temperature–duration experiment, mean LOI increased with increasing temperature but not exposure duration for both the 0- to 15- and the 15- to 50-cm samples (Table 2). Temperature was highly significant for both depths (F > 467, P < 0.0001), while duration was not (F < 2.3, P > 0.10). Pearson  $r^2$  values between LOI and SOC never exceeded 0.74, showed little consistent trend to change with increasing duration, and tended to be highest at 300°C.

After 15 min at 300°C, 79% of the 2-h LOI had already occurred for high-LOI aspen ecosystems, while 86% had occurred for low-LOI black cinder ecosystems (Fig. 1). After 90 min, 98 to 99% of the 2-h LOI had already occurred, on average, for all samples.

Regression equations estimating SOC from LOI (300°C, 2 h) using 100 samples for each depth attained  $r^2$  values of 0.69 (0– 15 cm) and 0.58 (15–50 cm; Fig. 2). The *y* intercepts were small, and forcing the regressions through the origins did not change the  $r^2$  values. Clay concentration was the only variable in addition to



Fig. 1. Cumulative loss-on-ignition through time for the same set of samples from northern Arizona ponderosa pine forest soils. Data are shown for 0- to 15-cm soils from aspen and black cinder ecosystem types (n = 3 each) at extremes of loss-on-ignition and as an overall mean consisting of three samples from each of 10 ecosystem types.



little after 2 h of heating British soils at 550°C. In contrast, Schulte et al. (1991) determined that LOI at 360°C differed significantly among 2-, 4-, and 16-h heating durations in two soils ranging in LOI from 4 to 46%. Ben-Dor and Banin (1989) reported intermediate results, with 80 to 90% of LOI at 400°C occurring within the first 8 h in arid Israeli soils. These variations in times to constant weight may result from differences in LOI temperature, organic contents, or other factors.

Consistent with previous studies (e.g., Ranney, 1969; Shulte and Hopkins, 1996), temperature was a dominant factor affecting LOI, with a near doubling of LOI when the temperature was increased from 300 to 600°C (Table 2). Morever,  $r^2$  values between LOI and SOC tended to decrease with increasing temperature. In one of the few previous studies examining temperature effects on LOI-SOC relationships, Donkin (1991) found that temperatures of 350 to 450°C produced the highest correlations among nine temperatures ranging from 150 to 900°C. He found, however, that  $r^2$  values were similar (0.92–0.94) for temperatures between 250 and 900°C. Temperature had a larger effect on LOI-SOC relationships in our study, with  $r^2$  values for 0-



LOI that improved SOC estimation in multiple regression. Clay slightly improved  $r^2$  by 0.09 to 0.78 (0–15 cm) and by 0.06 to 0.64 (15–50 cm). Including additional variables (sand, pH, or CaCO<sub>3</sub> equivalent) produced improvements of <0.02.

Relationships between LOI and SOC varied among ecosystem types. For example, 0- to 15-cm  $r^2$  values ranged from zero (xeric basalt ecosystems) to 0.97 (mesic limestone ecosystems). There were no clear trends, however, for relationships to be stronger or weaker among ecosystems with certain soil properties or parent materials. Basalt parent materials, for instance, exhibited the lowest  $r^2$  (xeric basalt ecosystems) and among the highest  $r^2$  (0.93, rocky basalt ecosystems). Half of the 10 ecosystem types for both depths exhibited  $r^2$  values  $\leq 0.7$ .

### DISCUSSION Heating Duration and Temperature

We did not find that heating samples at a given temperature for longer than 2 h increased LOI or consistently increased the  $r^2$  between LOI and SOC (Table 2). In fact, in our test at 300°C, we found that 85 to 89% of LOI occurred in the first 30 min and 98 to 99% occurred in the first 90 min (Fig. 1). Howard (1965), for example, also found that LOI changed to 15-cm samples differing by 0.18 to 0.24 when increasing temperature from 300 to 600°C (Table 2).

### Loss-On-Ignition–Soil Organic Carbon Relationships

Loss-on-ignition explained only 69% (0–15-cm samples) and 58% (15–50-cm samples) of the variation in SOC in our regression equations using 100 samples for each depth (Fig. 2). These relationships are weak compared with other published studies, which have reported  $r^2$  values >0.85 (Table 3). Clay concentration was the only variable in our study that improved predictions in multiple regressions, although improvements were only slight (6–9% increases in variability explained). While determining specific reasons why LOI–SOC relationships were weak is beyond the scope of this study, there are some possibilities. These weak relationships may be related to the LOI temperature–duration combinations we used, methodological problems in measuring both SOC and LOI, the fairly narrow range of SOC in our samples, inherent characteristics of the soils, or other factors.

A temperature and duration combination that we did not test could have resulted in stronger LOI–SOC relationships. We did test nine temperature-duration combinations,

Table 3. Summary of studies estimating soil organic C (SOC) from loss-on-ignition (LOI) at various heating temperatures and durations. Only studies that directly measured SOC (e.g., using an elemental analyzer) are included.

Tomporaturo	Duration	SOC = mLOI + b			SOC range	Lavor	Poforonco		
remperature	Duration	т	b	<i>r</i> <sup>2</sup>	- SOC Tallge	Layer	Kelerence		
°C	h				% wt.				
300	2	0.4315	0.1603	0.69	<1–5	0–15 cm	This study		
300	2	0.3410	0.0322	0.58	<1-3	15–50 cm	This study		
600	6	0.405	-0.710	0.86	2-6	0–20 cm	Goldin (1987)		
450	4	0.5318	-6.9917	0.92	<1-3	0–8 cm	Cambardella et al. (2001)		
405	12	0.520	-0.0134	0.92	<1-9	0–25 cm	David (1988)		
360	2	0.6824	-2.8696	0.97	<1-6	A horizon	Konon et al. $(2002)$ +		
360	2	0.6094	0.1949	0.98	<1-12	A horizon	Kohen et al. (2002)1		
375	16	-#	_	0.97	<1-8	0–33 cm	Soon and Abboud (1991)		
550	4	0.5076	-0.2284	0.99	<1-52	Oi–210 cm	Christensen and Malmros (1982)		
550	CW§	0.5398	-0.9888	0.99	5-50	10–15 cm	Howard (1965)		
450	16	0.453	0.015	0.99	<1-5	0–15 cm	Lowther et al. (1990)		
≤375¶	28¶	0.5556	-0.1944	0.99	1–45	A + B horizons	Ranney (1969)		

+ This study included equations developed separately for five different major land resource areas (MLRAs). Equations for two representative MLRAs (103 and 108) are shown.

 $\pm$  The estimation equation reported for this study was as follows: SOC = 0.633(  $\pm$  0.008)LOI - 9.36(  $\pm$  0.54).

§ Constant weight.

¶ Ranney's (1969) procedure included low-temperature (100–200°C) ashing for 4 to 5 d and heating in a muffle furnace at 375°C for 28 h.

however, spanning ranges commonly used in LOI studies (Schulte and Hopkins, 1996). It is possible that temperatures <300°C might result in stronger relationships with our soils, because there was a slight trend for LOI–SOC relationships to weaken with increasing temperatures (Table 2).

Methodological problems, both with the SOC elemental analysis and the LOI procedures, might have decreased accuracy. However, we followed standard procedures for both analyses (Nelson and Sommers, 1996; Shulte and Hopkins, 1996). Furthermore, repeated measurement errors for both SOC and LOI averaged <5%, and no problems were evident in analyses of blanks and laboratory reference samples using the elemental C analyzer. Nevertheless, it is difficult to entirely rule out the possibility of some unknown methodological problem. It also is possible that processing samples and performing analyses differently, such as grinding LOI samples more finely than just to pass a 2-mm sieve, might have changed results. Shulte and Hopkins (1991) found that LOI was insensitive to such factors as the number of samples placed in a muffle furnace at one time, but that the weight of samples used did affect LOI. It is unclear whether sample weight affects LOI-SOC correlations.

Our samples did contain a fairly narrow range of SOC, with maximum SOC concentrations on the low end of those reported in LOI studies (Table 3). Donkin (1991) found that LOI–SOC relationships were strongest in soils with low (<5%) SOC concentrations. Conversely, Soon and Abboud (1991) concluded that LOI was unreliable for estimating SOC in low (<1.5%) SOC soils. We detected no consistent trend for LOI–SOC relationships to strengthen for ecosystems high or low in SOC. Additionally, slopes and y intercepts for our LOI–SOC regressions are not unusual compared with other studies (Fig. 2, Table 3).

There might be inherent characteristics of soils in our study area that weakened LOI–SOC relationships. The presence of carbonates has sometimes been cited as impacting LOI–SOC relationships, but typically only when using LOI temperatures above approximately 500°C (e.g., Davies, 1974). Few of our samples contained carbonates (Table 1), however, and we also tested two temperatures below 500°C. Loss of minerals or structural water from clay also have been implicated as affecting LOI–SOC relationships (Ball, 1964; Spain et al., 1982), but inclusion of clay improved estimation of SOC by <10% in our multiple regressions. Fires historically burned frequently across our study area (Covington et al., 1997), and it is possible that charcoal or elemental C remaining in soils from these fires (DeLuca et al., 2006) affected LOI–SOC relationships. Our study area also is semiarid, and few LOI studies have been performed in semiarid or arid soils. Ben-Dor and Banin (1989), however, reported strong LOI–SOC relationships in arid Israeli soils with SOC estimated using the Walkley–Black procedure.

Other factors cannot currently be ruled out for explaining our weaker LOI–SOC relationships compared to other studies. It is possible that other studies have found weak LOI–SOC relationships, but analogous to underpublishing of statistically nonsignificant results (Møller and Jennions, 2001), these results have not been published. As another example,  $r^2$  values might not be optimal for assessing the ability of LOI to estimate SOC and for comparisons among studies (Neter et al., 1996; Harris et al., 2001). Our LOI–SOC regression equations did exhibit wide scatter (Fig. 2), however, and a purpose of LOI–SOC research is to develop simple, readily useable LOI–SOC equations (Konen et al., 2002).

### CONCLUSIONS

Our data suggest that LOI may be useful for estimating SOC in these soils when only rough estimates are required. Future research could try to clarify reasons why our LOI–SOC relationships were weak compared with other published studies. Modifications to the standard LOI procedure used here, such as more finely grinding samples, also could be tested for improving relationships with SOC. Our results suggest, however, that directly measuring SOC, such as with an elemental C analyzer, may be more accurate and straightforward than estimating SOC from LOI in this region.

### ACKNOWLEDGMENTS

We thank Steve Overby, Lauren Hertz, and the U.S. Forest Service, Rocky Mountain Research Station Flagstaff Analytical Lab for use and operation of their C analyzer. This work was supported by the Ecological Restoration Institute and the School of Forestry at Northern Arizona University. We also appreciate comments from Mark Johnson and two anonymous reviewers on an earlier draft of the manuscript.

### REFERENCES

- Abella, S.R., and W.W. Covington. 2006. Forest ecosystems of an Arizona *Pinus ponderosa* landscape: Multifactor classification and implications for ecological restoration. J. Biogeogr. 33:1368–1383.
- Alexander, L.T., and H.G. Byers. 1932. A critical laboratory review of methods of determining organic matter and carbonates in soil. USDA Tech. Bull. 317. U.S. Gov. Print. Office, Washington, DC.
- Allen, C.D., M. Savage, D.A. Falk, K.F. Suckling, T.W. Swetnam, T. Shulke, P.B. Stacey, P. Morgan, M. Hoffman, and J.T. Klingel. 2002. Ecological restoration of southwestern ponderosa pine ecosystems: A broad perspective. Ecol. Appl. 12:1418–1433.
- Ball, D.F. 1964. Loss-on-ignition as an estimate of organic matter and organic carbon in non-calcareous soils. J. Soil Sci. 15:84–92.
- Barnes, B.V., K.S. Pregitzer, T.A. Spies, and V.H. Spooner. 1982. Ecological forest site classification. J. For. 80:493–498.
- Ben-Dor, E., and A. Banin. 1989. Determination of organic matter content in arid-zone soils using a simple "loss-on-ignition" method. Commun. Soil Sci. Plant Anal. 20:1675–1695.
- Broadbent, F.E. 1953. The soil organic fraction. Adv. Agron. 5:153-183.
- Cambardella, C.A., A.M. Gajda, J.W. Doran, B.J. Wienhold, and T.A. Kettler. 2001. Estimation of particulate and total organic matter by weight losson-ignition. p. 349–359. *In* R. Lal et al. (ed.) Assessment methods for soil carbon. Lewis Publ., New York.
- Christensen, B.T., and P.A. Malmros. 1982. Loss-on-ignition and carbon content in a beech forest soil profile. Holarctic Ecol. 5:376–380.
- Covington, W.W., P.Z. Fulé, M.M. Moore, S.C. Hart, T.E. Kolb, J.N. Mast, S.S. Sackett, and M.R. Wagner. 1997. Restoring ecosystem health in ponderosa pine forests of the Southwest. J. For. 95:23–29.
- Dane, J.H., and G.C. Topp (ed.). 2002. Methods of soil analysis. Part 4. Physical methods. SSSA Book Ser. 5. SSSA, Madison, WI.
- David, M.B. 1988. Use of loss-on-ignition to assess soil organic carbon in forest soils. Commun. Soil Sci. Plant Anal. 19:1593–1599.
- Davies, B.E. 1974. Loss-on-ignition as an estimate of soil organic matter. Soil Sci. Soc. Am. Proc. 38:150–151.
- DeLuca, T.H., M.D. MacKenzie, M.J. Gundale, and W.E. Holben. 2006. Wildfire-produced charcoal directly influences nitrogen cycling in ponderosa pine forests. Soil Sci. Soc. Am. J. 70:448–453.
- Donkin, M.J. 1991. Loss-on-ignition as an estimator of soil organic carbon in A-horizons of forestry soils. Commun. Soil Sci. Plant Anal. 22:233–241.

- Goh, T.B., R.J. St. Arnaud, and A.R. Mermut. 1993. Carbonates. p. 177–185. In M.R. Carter (ed.) Soil sampling and methods of analysis. Lewis Publ., Boca Raton, FL.
- Goldin, A. 1987. Reassessing the use of loss-on-ignition for estimating organic matter content in noncalcerous soils. Commun. Soil Sci. Plant Anal. 18:1111–1116.
- Gundale, M.J., T.H. DeLuca, C.E. Fiedler, P.W. Ramsey, M.G. Harrington, and J.E. Gannon. 2005. Restoration treatments in a Montana ponderosa pine forest: Effects on soil physical, chemical and biological properties. For. Ecol. Manage. 213:25–38.
- Harris, I.R., B.D. Burch, and R.T. St. Laurent. 2001. A blended estimator for a measure of agreement with a gold standard. J. Agric. Biol. Environ. Stat. 6:326–339.
- Howard, P.J.A. 1965. The carbon–organic matter factor in various soil types. Oikos 15:229–236.
- Keeling, P.S. 1962. Some experiments in the low-temperature removal of carbonaceous material from clay. Clay Mineral. Bull. 28:155–158.
- Konen, M.E., P.M. Jacobs, C.L. Burras, B.J. Talaga, and J.A. Mason. 2002. Equations for predicting soil organic carbon using loss-on-ignition for north central U.S. soils. Soil Sci. Soc. Am. J. 66:1878–1881.
- Lowther, J.R., P.J. Smethurst, J.C. Carllyle, and E.K.S. Nambiar. 1990. Methods for determining organic carbon on podzolic sands. Commun. Soil Sci. Plant Anal. 21:457–470.
- Miller, G., N. Ambos, P. Boness, D. Reyher, G. Robertson, K. Scalzone, R. Steinke, and T. Subirge. 1995. Terrestrial ecosystems survey of the Coconino National Forest. U.S. For. Serv., Southwest. Region, Albuquerque, NM.
- Møller, A.P., and M.D. Jennions. 2001. Testing and adjusting for publication bias. Trends Ecol. Evol. 16:580–586.
- Nelson, D.W., and L.E. Sommers. 1996. Total carbon, organic carbon, and organic matter. p. 961–1010. *In* D.L. Sparks (ed.) Methods of soil analysis. Part 3. Chemical methods. SSSA Book Ser. 5. SSSA, Madison, WI.
- Neter, J., M.H. Kutner, C.J. Nachtsheim, and W. Wasserman. 1996. Applied linear statistical models. Irwin Publ., Chicago, IL.
- Ranney, R.W. 1969. An organic carbon–organic matter conversion equation for Pennsylvania surface soils. Soil Sci. Soc. Am. Proc. 33:809–811.
- SAS Institute. 2002. JMP version 5 user's guide. SAS Inst., Cary, NC.
- Schulte, E.E., and B.G. Hopkins. 1996. Estimation of soil organic matter by weight loss-on-ignition. p. 21–31. In F.R. Magdoff et al. (ed.) Soil organic matter: Analysis and interpretation. SSSA Spec. Publ. 46. SSSA, Madison, WI.
- Schulte, E.E., C. Kaufmann, and J.B. Peter. 1991. The influence of sample size and heating time on soil weight loss-on-ignition. Commun. Soil Sci. Plant Anal. 22:159–168.
- Soon, Y.K., and S. Abboud. 1991. A comparison of some methods for soil organic carbon determination. Commun. Soil Sci. Plant Anal. 22:943–954.
- Spain, A.V., M.E. Probert, R.F. Isbell, and R.D. John. 1982. Loss-on-ignition and the carbon contents of Australian soils. Aust. J. Soil Res. 20:147–152.
- Sparks, D.L. (ed.). 1996. Methods of soil analysis. Part 3. Chemical methods. SSSA Book Ser. 5. SSSA, Madison, WI.